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THE CHEMISTRY OF THE CLAY-SIZE FRACTION
ACROSS THE OLDMAN-BEARPAW CONTACT OF
THE ST. MARY RIVER SECTION

R.N.FARVOLDEN

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THE CHEMISTRY OF THE CLAY - SIZE FRACTION ACROSS THE OLDMAN-BEARPAW CONTACT OF THE ST. MARY RIVER SECTION

A DISSERTATION
SUBMITTED TO THE SCHOOL OF GRADUATE STUDIES
IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE
OF MASTER OF SCIENCE

FACULTY OF ARTS AND SCIENCE DEPARTMENT OF GEOLOGY

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EDMONTON, ALBERTA March 27, 1957



Abstract

The clay-sized fraction of samples collected across the Oldman-Bearpaw contact of the St. Mary River section were analysed. The $\mathrm{Na_2O}$, $\mathrm{K_2O}$, $\mathrm{P_2O_5}$ and total iron analyses show a break at the contact between the non-marine and marine beds. Other predicted clay chemistry changes do not occur in this section because the normal clay diagenesis was masked and the system was upset by fall-out of volcanic ash.

The investigation shows that the method of rapid silicate analysis can be used for certain geologic studies but the results are not as reliable as those obtained by conventional methods.

Acknowledgments

This investigation was made possible through the co-operation of the University of Alberta and the Research Council of Alberta in pooling equipment, chemical supplies and facilities. Many staff members of both organizations offered encouragement and useful counsel and the help of Drs. C. P. Gravenor, J. D. Godfrey and P. J. S. Byrne of the Research Council of Alberta and Drs. R. E. Folinsbee and C. R. Stelck of the University of Alberta is particularly acknowledged.

Mr. H. Wagenbauer, Research Council of Alberta laboratory technician, aided in preparation and analysis of the samples.

Mr. J. Groot, draftsman-compiler for the Research Council of Alberta, gave advice and help in the preparation of the figures.



 $\label{eq:shellout} Financial \ assistance \ was \ received \ through \ the \ Shell \ Oil \ Fellowship,$ awarded to the author.



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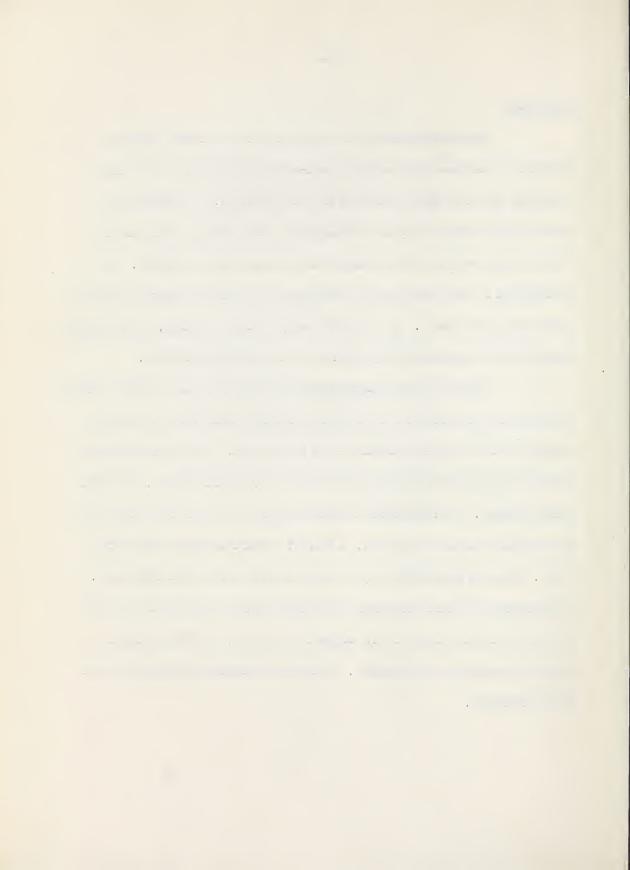
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Introduction

Sedimentologists have long been prone to give careful field and laboratory descriptions of the sand- and silt-sized fractions of the clastic rocks and ignore all but the gross features of clay or shale rocks. This tendency is understandable since the particles and crystals of clay minerals which form the shales are too small to yield to thorough study by conventional methods. The development of x-ray equipment and techniques has enabled scientists to partially break through this barrier. As a result the study of clay mineralogy, and the origin, deposition and occurrence of clay minerals has received wide attention.

It has been demonstrated that the majority of the clay mineral material in sediments is the product of the chemical weathering of silicate rocks and the adjusting of this weathered product to local environment. The adjustment involves chemical exchanges between the clay minerals and the water medium, and minor lattice changes. The regeneration of degraded illite and chlorite takes place while the sediment is still in suspension, or while it is near the surface of the bottom muds. Some montmorillonite may be altered to illite at this same early stage. The formation of chlorite from illite and degraded illite is a function of time and salinity and occurs during the early stages of diagenesis, as well as during the stages of suspension and deposition. The time of alteration of kaolinite has not been established.



There is good evidence to show that both the chemical and the lattice alterations occur in a predictable pattern in present day sediments. This investigation was conducted to determine whether or not the chemical changes are preserved and detectable in ancient sediments.

In this investigation the clay-size fraction of shale samples taken across the Oldman-Bearpaw contact of the St. Mary River section were analysed in an effort to determine what clay-chemistry changes were present.

The Oldman formation in this area is mostly sandy shale and is classified on the basis of fauna and flora as a continental deposit. The formation contains coal seams, plant fragments and fresh water pelecypod shells. The Bearpaw formation is almost exclusively greyish brown shale in the lower part of the section, and contains ammonite and marine pelecypod remains.

 $\mbox{\sc J.}$ H. Wall found freshwater ostracods in the Oldman samples and a marine microfauna in the Bearpaw samples.

The "Rapid Analysis" methods of Shapiro and Brannock (1956) were followed except in the determination of Al_2O_3 , organic matter and $H_2O \cdot Al_2O_3$ was determined by the method outlined by M. L. Jackson 1 . Total loss on ignition was considered to include organic matter, crystalline and adsorbed water, and sulfur.

Professor of Soils, University of Wisconsin. Details of the method were obtained from Jackson's lecture notes on soil analysis, which are to be published.

Clay Minerals in the Sedimentary Cycle

Weathering

Reiche (1945) has defined weathering as follows: "the response of materials which were in equilibrium within the lithosphere to conditions at or near its contact with the atmosphere, hydrosphere and perhaps still more importantly, the biosphere".

The chemical and physical changes which occur in weathering or soil-forming processes are of prime geologic interest since qualitatively and quantitatively the material available to erosion, transport and deposition is dependent on these changes. The sequence of these changes in the breakdown of a rock and the formation of new minerals will depend upon conditions prevailing at the place of weathering. The weathered zone may be wet or dry, hot or cold, covered with vegetation or barren, poorly drained or well drained and each of these conditions may be continuous or discontinuous. Thus there can be a great variation in the pH, eH, leaching, and mechanical stress conditions acting on the rock materials present.

The occurrence of clay minerals as a weathered product of sedimentary rocks can be easily explained. Simple solution of the cement of the host rock and mechanical breakdown could give a soil rich in clay minerals.

In the weathering of igneous and metamorphic rock types, composed mainly of silicate minerals, mechanical breakdown and removal of soluble ions

will not produce clay minerals. Except for the micas, the crystal lattices of the silicate minerals must be broken down and rebuilt as phyllosilicate structures. This can be accomplished only by chemical reaction in the soil profile and mechanical weathering is only important in that it increases the surface area, thus facilitating chemical activity.

Silicate minerals are practically insoluble in water so processes other than solution are responsible for their breakdown according to Keller (1957). The most significant of these processes are hydrolysis and ion exchange, chelation, dialysis, solution, carbonation and hydration. The mechanisms of each of these reactions is such that within the environment provided by the weathering zone they may act upon any of the silicate structures, adding, exchanging, or removing ions, weakening bonds and causing lattice destruction.

Hydration and hydrolysis are likely the most important of these processes. Under most conditions the silicate structure which yields most easily to weathering processes is that which is composed of independent or incompletely linked silica tetrahedra, the nesosilicates and the sorosilicates. Minerals possessing these structures are olivine and beryl. These structures are susceptible to hydrolysis of the strong base which joins silica tetrahedra. The OH ion of water can unite with the cation of the mineral to form a hydroxide and the H ion then replaces the cation in the crystal structure. The concentration of charge in the small volume of the H ion upsets the lattice and initiates its destruction. Inosilicates such as amphiboles and pyroxenes, and some phyllosilicates break down in the same manner. This

hydrolysis of the strong base joining structural units is really a form of cation exchange with the H ion replacing the cation of the mineral.

The destruction of the feldspar lattice can be accomplished by the chemical activity of pure water. According to Frederickson (1951) water, adsorbed onto the surface of albite, may be crystalline in form. The exterior oxygens of the albite crystal are electrically deficient in protons because they have only one valence satisfied, the other bond being terminated by the surface of the crystal. The molecules of HOH are polarized. The negative charges of the oxygen are located on one side of the atom subtending an angle of 120 degrees while the hydrogen ions are grouped on the other side of the oxygen atom. The positive side of the HOH molecule will thus be strongly attracted to the negative surface of oxygens of the albite crystal, and the hexagonal pattern formed by the HOH lattice fits that of the oxygen atoms of the tectosilicate structure.

Physiochemical evidence indicates that the first layer of HOH molecules adsorbed is very strongly attached to the silicate structure. These molecules are all oriented with their positive side toward the silicate surface, so the negatively-charged surface of the crystal is reproduced by this first water layer. By the same mechanism other non-liquid water layers may be adsorbed with the positive side attracted to a negative surface until, gradually, thermal agitation becomes a disrupting factor and the HOH progressively loses its non-liquid, or crystalline character.

At the interface of the crystal and the first adsorbed water layer, the oxygen of the mineral has one unsaturated negative charge and the oxygen of the water has two hydrogen ions. One of these hydrogens can satisfy the oxygen of the mineral and the other is surplus. Within the albite lattice the sodium ion is positioned in six-fold symmetry satisfying the surplus negative charge which has resulted from aluminum replacing silicon. The sodium is loosely held as its charge is shared by six oxygens. At any point in its structure where two oxygens are touching, each one has a one-sixth negative charge compensated by a relatively distant sodium ion. The surplus hydrogen ion at the crystal-water interface will migrate into the lattice and take up a position where these two oxygens touch. The result is an excess positive charge in the lattice and, as the hydrogen ion is more firmly held than the sodium, due to concentration of charge and proximity of negative units, the sodium ion will be expelled from the crystal lattice.

The ionic environment outside the mineral lattice is much the same as within it due to the orientation of the HOH molecules so the sodium ion diffuses down gradient from high to low sodium concentration.

In the silicate lattice the oxygens opposite the immigrant hydrogen ion are left with unsatisfied negative charges and these repel each other, expanding the lattice and weakening it. Lattice breakdown by this method could be applied to any feldspar.

This reaction depends upon removal of the strong base from the system which might be accomplished by dialysis, chelation, adsorption on another crystal

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fragment or simple solution. The lattice debris of silicon-aluminum-oxygen atoms may be removed or it may be reconstituted in situ as a clay mineral. The reaction, product and by-products will be dependent upon the local conditions of pH, eH, permeability and temperature and so in their generation clay-minerals are seen to be very susceptible to geochemical adjustment to environment.

Jackson et al (1948) attempts to show that there is a regular reaction series with progressive weathering and leaching and the soil mineral content will therefore indicate the "stage" of weathering. He holds that the end products will be hematite, gibbsite and anatase regardless of the parent material, but recognizes that reversals in development can take place with slight changes in environment. For example, where strong bases are being removed in water solution, silica is comparatively soluble and can also be removed. Where the solutions are acid silica is almost insoluble.

Other workers believe that the composition of the parent material dominates the weathering reaction and products. Cady (1950) shows that the soil in an area in North Carolina still reflects the underlying lithology although the area is chronologically and morphologically old. Kerr (1952) cites Hoskings' work in Australia where the clay mineralogy in soils is dependent upon parent rock and local environment.

There is good evidence to indicate that where conditions are favorable and stable for long periods of time, the weathering process does follow a series of stages leading to laterization. In most soils the process is interrupted by

erosional, climatic or drainage conditions and an "immature" clay assemblage is the product of the weathering.

Transportation

The effects of soil -, vadose -, and groundwaters on the exposed rock must be considered as part of the weathering process but the leaching effect of surface waters is more suitably termed erosion. Potassium, sodium, magnesium and calcium and their common anions CO_3^{--} , SO_4^{--} , NO_3^{--} and Cl^- , are removed in solution. The solution may occur during surface wash or as part of the soil-forming process. In the latter case the ions may be carried into the surface drainage system by springs or seepage. Some of the dissolved salts of rivers is added by the addition of fine detritus from the weathered zone. This fine detritus must adjust to a changed environment and loosely held ions may be taken into solution, where perhaps due to restricted permeability they had been incompletely leached in the solum.

Very small amounts of silica can be carried in true solution where the pH is neutral to basic. However, these small quantities will not account for the fact that silica comprises over eleven per cent of the dissolved and colloidal load of the average river (Clarke, 1924).

Silica is known to occur in the weathered zone in colloidal-size fragments (Murray and Gravenor, 1953) and these particles become part of a river load of sediment. In alkaline soils, in the presence of sodium and potassium ions silica is relatively soluble. This dissolved silica might be present in the form of complex

ions or neutral molecules. When these solutions come into contact with river waters with lower pH and lower alkali concentration the complex ions and neutral molecules (H_4SiO_4) may polymerize, expelling cations and leaving a network of silica tetrahedra in a colloidal suspension. Correns (1940), as quoted by Taylor (1952) believes that SiO_3 ions polymerize with aging to form a colloid while Roy (1945) holds that silica is in true solution in natural waters.

Aluminum is practically non-existent in natural surface waters in either the dissolved or colloidal fraction. At the pH of surface waters aluminum is insoluble (practically speaking) and its hydroxide is unstable in a water medium. However, the element is transported somehow from the site of weathering to that of deposition, likely as a component of the degraded, leached clay fragments and "lattice debris" in the suspended sediment.

A number of relatively insoluble oxides form in the soil profile and these are transported as colloids, in the form of hydroxides or hydrated oxides.

Important examples are, ferric and ferrous hydroxide, chromium hydroxide, and the hydrated dioxides of titanium, thorium, zirconium, vanadium and manganese.

Ferrous iron is present in some streams in true solution.

According to most recent reports the majority of the clay-sized fraction in the suspended sediment is composed of montmorillonite, illite and kaolinite.

Montmorillonite and illite are formed early in the weathering process and where erosional conditions don't permit a mature soil to form, they will be dominant over kaolinite. The illite is often "degraded", the interlayer potassium and surface

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adsorbed cations having been removed by leaching. Even octahedral cations may have been removed with only a residual, negatively charged, relic structure remaining. Kaolin may comprise an important part of the sediment where the drainage basin includes areas which are geomorphologically mature, but may be absent in sediment derived from areas undergoing severe erosion.

These then are the materials present in surface waters which are of importance to clay genesis and diagenesis. The pH is normally between six and eight.

Deposition

When the river water enters the ocean the environment changes and the material introduced tends to undergo geochemical adjustment toward equilibrium with this environment.

The soluble salts are readily acceptable by the marine waters because, except for localized areas, the sea is unsaturated in dissolved salts. Dissolved silica is notably scarce in marine waters which would seem to indicate that the colloidal and/or dissolved silica of the fresh water is flocculated immediately on contact with ocean brines of high electrolytic content. The flocculated silica could settle to the floor of the ocean in layers or in colloid-size clumps and with aging become chert. In the same way the iron hydroxides might form a bedded iron deposit or ferruginous shales. Where this silica and iron is flocculated with the suspended clay sediments, it could be incorporated into bottom muds as part of a clay lattice or as colloid-size aggregates within a matrix of clay sediment.

 Siever (1957) holds that most silica is removed from the marine waters by organisms.

The clay-mineral colloids and the fresh water medium are suspended at the surface due to density and agitation factors and may move toward the open sea as a stream of fresh water on top of the salt water (Powers, 1953).

The influence of the electrolyte causes the clay colloid clumps to flocculate until a critical size is reached and they sink. The small size fraction still suspended at the surface is diluted by this loss. The chances of collision between colloidal particles in the dispersed phase are thereby decreased and the particles may remain suspended until agitation is so slight they can sink by gravity. This delay in settling allows the particles opportunity to develop in size and crystal form, a feature noted by Powers (1957) in the Chesapeake Bay area.

Studies of the bottom sediment from various deltaic, estuarine, off-shore and deep sea environments have been reported by recent investigators. Although local variations in clay mineralogy have not been adequately explained, a pattern is obvious. In nearly all cases the clay minerals which have been leached and degraded in a continental environment tend to pick up cations and recrystallize in a marine environment.

Thus, off the coast of California, Grim, Deitz and Bradley (1949) found that bottom samples all contained illite, montmorillonite, and kaolinite.

Illite was the most abundant and kaolinite the least abundant, in general. Complex

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crystalline and mechanical mixtures were common. In this case the Colorado River is the transporting agent and kaolinite is abundant in the sediment of the river and in the near-shore clays. Kaolinite content decreases rapidly off-shore due to diagenetic alteration to illite and chlorite. Consider the change from kaolinite to illite. The kaolin must add a second silica layer to its lattice and exchange aluminum for silicon in the tetrahedral layer and magnesium, titanium, iron, manganese or lithium for aluminum in the octahedral layer. In the latter change, aluminum is not necessarily expelled from the structure. The added cations may occupy octahedral positions left vacant in a dioctahedral structure. The unbalanced negative lattice is satisfied by adsorption of potassium ions on the basal planes of the clay lattice. A layer of brucite may be acquired resulting in chlorite being the end product.

The montmorillonite content was found to be fairly uniform off the mouth of the Colorado River showing that any alteration is slow. Thermal analysis of the samples suggests that montmorillonite content decreases slightly away from shore. This decrease could be due to alteration to chlorite or illite. In near-shore core samples the K_2O content of the clays increases with depth. Potassium is being taken out of the water by the formation of illite but this change doesn't keep up with sedimentation. Farther from the shore the K_2O content does not change with depth so illite formation there keeps up with sedimentation. The off-shore samples have a slightly higher MgO content than near-shore samples

and MgO increases with depth of core indicating that magnesium must be added slowly to clays during diagenesis. Further proof of diagenetic change lies in the observed change in the ratio of ${\rm SiO_2/Al_2O_3}$ in near-shore and off-shore clays. The near-shore kaolin-rich sediments have a lower ${\rm SiO_2/Al_2O_3}$ ratio than the kaolin-poor off-shore sediments. The ratio off shore is such that free silica is surely present.

The clay minerals in the sediments of the lower Guadalupe River and adjacent marine environments have been studied by Grim and Johns (1953). The river sediment is predominantly montmorillonite, with minor illite, kaolinite and chlorite. On entering the Gulf of Mexico the same pattern of change as observed on the investigation off the California coast (above) takes place. However, in this case most montmorillonite undergoes an initially rapid alteration to chlorite and illite and then the reaction rate slows down but maintains the same direction.

These lattice changes are accompanied, as before by the appropriate chemical changes. The $\rm K_2O$ and MgO content of the clay fraction increases from the delta towards the open water, and since the $\rm Al_2O_3/K_2O$ and $\rm Al_2O_3/MgO$ ratios decrease at the same time, the added cations are contributing to a new clay structure. At the delta the Na/Mg and Na/K ratios in the water increases more than can be accounted for by dilution alone. This is the result of the extraction of magnesium and potassium from the water accompanying the formation of illite and chlorite. All the montmorillonite cannot be converted since it is buried too quickly. Kaolinite is present in the fresh river water but not the marine sediments. Its absence there is due to alteration to chlorite. The clay mineral composition does not change with depth in core samples so major diagenetic changes are

contemporaneous with deposition. Samples from Barrier Island, which is flooded at high tide, have the highest content of illite and the lowest content of montmorillonite of any samples tested. Repeated wetting and drying favors the alteration. It was noted that crystals of the clay minerals in this basin are mostly less than 0.1 micron. In ancient clays the crystals are larger so diagenetic and post diagenetic conditions favor crystal growth.

Powers (1957) found a similar clay conversion occurring in the Chesapeake Bay area. The sediment of the Patuxent River is mainly illite and degraded illite with minor kaolinite and a trace of chlorite. The chlorite content shows a steady increase at the expense of degraded illite as the salinity of the water increases in the estuary. The effect of time is not as great as that of salinity but core samples show an increase in chlorite and a decrease in degraded illite with increasing age of the sediment. This information from x-ray analysis has been substantiated by the change in the chemistry of the interstitial water. Magnesium concentration in the interstitial water decreases with depth (and time) and the potassium concentration increases giving evidence that magnesium is adsorbed in preference to potassium.

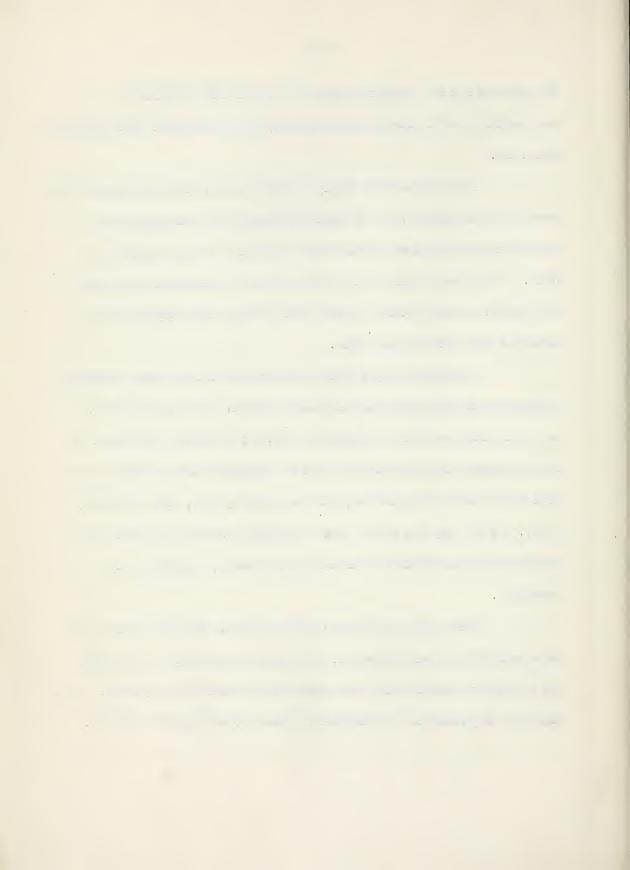
The effect of time seems to be more important than the effect of salinity in the alteration of clays in the Mississippi Delta area. Milne and Earley (1958) found that only minor changes occur when the clay minerals of the Mississippi sediments enter the Gulf of Mexico. However, a core taken near the shelf edge region, several miles from the mainland, contained a much higher proportion of illite than the bottom muds near the mouth of the Mississippi River.

It is believed that the high illite content is the result of the alteration of montmorillonite and that the sediment originally had a much higher montmorillonite-illite ratio.

In the Neuse River estuary in North Carolina, Griffin and Ingram (1955) have shown that kaolinite, the dominant clay mineral being introduced by the river, decreases downstream in the estuarine deposits in favor of chlorite and illite. A considerable portion of the river sediment is amorphous to x-rays and they suggest that this material is lattice-debris which is reconstituted to form chlorite or illite in the marine waters.

According to Taylor (1952) and Dietz (1941), ocean bottom sediment is predominantly illite with kaolinite generally present, especially near shore, and montmorillonite of little significance except in the vicinity of volcanoes. It seems anomalous that kaolinite should not be completely altered to illite or some other hydrous mica as this tendency has been proved by Grim, Dietz and Bradley (1949). Perhaps only near shore is there enough SiO₂ available in the water for the kaolinite to accumulate the extra silica tetrahedral layer required for the conversion.

Millot (1952) and Murray (1953) both believe that clay minerals will adjust according to their environment. They carry the argument one step further and show that the resulting clay assemblage will be preserved in clay rocks. Millot states that clay rocks are "an authigenic rock from altered allogenic material".



He believes that the shales of the Paris Basin show by their clay mineralogy their original environments of deposition. The continental shales are high in kaolinite and the marine shales mainly composed of three-layered minerals. According to Murray, shales of the Pennsylvanian cyclothems of Indiana and Illinois have a clay assemblage which indicates the environment of deposition. As in the Paris Basin, the sedimentary facies has been determined by conventional methods. By x-ray analysis Murray shows that the illite content of the shales increases at the expense of kaolinite going from a non-marine, through a brackish to a marine environment.

Weaver (1958) shows that the composition of clay minerals in a sedimentary basin is determined mainly by the character of the source material. He has good evidence to prove that any common clay mineral species can be dominant in a shale, regardless of the environment of deposition of that shale. Weaver believes that the only diagenetic change which clay undergoes is a cation exchange and he does not accept any evidence which suggests that the clay crystal-lattice is changed during diagenesis.

The weight of evidence indicates that the diagenetic change does take place when clay minerals are introduced into a marine environment but .
this change does not go to completion.

Most of the authors mentioned offer explanations of the mechanics of these alterations but it is only the alterations that are pertinent to this thesis.

Geologic Setting

Upper Cretaceous time is represented on the plains region of Alberta by a sequence of shales, sandy shales and argillaceous sandstones and siltstones. The Oldman formation and the Bearpaw shale form part of this sequence. The ammonite bearing Bearpaw shale was deposited over the continental and brackish sandstones, shales and coals of the Oldman formation during the last marine invasion. It was felt that the two formations were a good example of a marine-non-marine sequence in sedimentation, and a section across the contact should therefore have a clay mineral assemblage to substantiate the evidence outlined above. This clay mineral assemblage should be reflected in the chemistry of the clay-sized fraction.

The Oldman Formation

The Oldman formation was named by Russell and Landes (1940) for the upper part of Dawson's Belly River series (Dawson, 1875). The formation is composed of fine sandstones and sandy shales, often bentonitic. From a distance the untcrops appear to be light grey sandstones but this is often due to surface wash and it is likely that argillaceous types predominate in the Oldman section. Lenticular beds of coarse clean sandstone occur and are commonly cross-bedded.

The Oldman formation carries terrestrial, fresh-water, and brackish water fauna and is generally accepted by geologists as being a continental deposit.

The Belly River series, of which the Oldman formation is the uppermost division,

was likely deposited in streams and lakes and marginal swamps during a withdrawal of the Pierre Sea. The abundance of dinosaur bones in the Oldman formation in southeastern Alberta is good evidence of the nature of the environment of deposition.

The upper part of the Oldman formation is composed of dark grey carbonaceous shales which in the Lethbridge area carry minable coal seams.

This horizon is known as the Lethbridge member and marks a lowering of the land surface with widespread marginal swamp development.

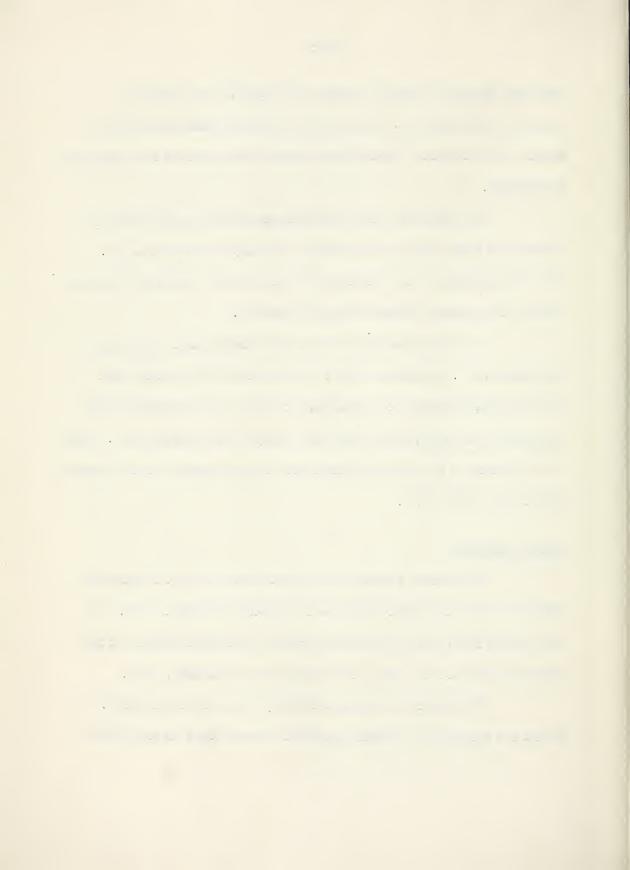
The Oldman formation is the surface formation over wide areas in the Alberta plains. It thickens toward the southwest and in the disturbed belt of the Cordillera it becomes the upper part of the thick and massive Belly River sandstone and is not distinguished from the underlying Foremost formation. Where it is encountered in the subsurface further north it is also grouped with the Foremost under the name Belly River.

Bearpaw Formation

The Bearpaw formation was deposited during the last transgression of the Pierre Sea during Upper Campanian time (Warren and Stelck, 1958). The sea advanced rapidly across the Oldman surface in southeastern Alberta but this advance was slower to the west of the Sweetgrass arch (Crockford, 1949).

The formation is almost exclusively shale in the lower portion.

Several thin siltstones and sandstones raised to member status are found in the



upper two-thirds of the formation (Link and Childerhose, 1931).

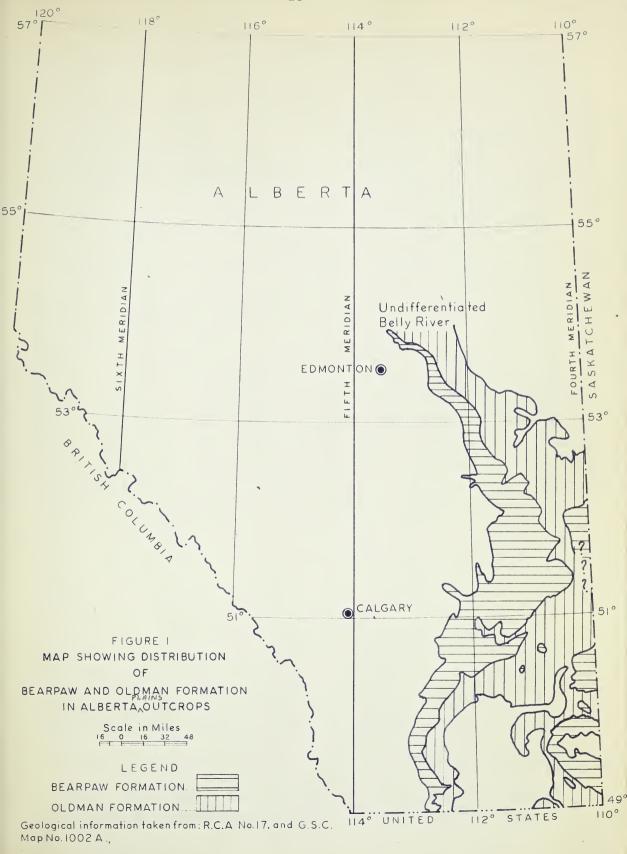
The shale is commonly dark grey and brownish grey and weathers to a rusty brown color. Link and Childerhose (1931) report that in fresh cores the formation is greyish green and that these cores weather in a few months to a brownish color. Abundant gypsum crystals occur on the outcrops. Discontinuous layers of spheriodal ironstone concretions are common and these concretions sometimes contain marine fauna. Bentonite beds, mostly less than a foot in thickness, occur throughout the formation. Both the concretionary layers and the bentonite beds are individually of limited extent and are not reliable for structure surveys. This lack of reliable horizons makes structure surveys difficult on outcrops of both the Oldman and Bearpaw formations.

The distribution of these two formations in Alberta outcrops is given in figure 1. Figure 2 shows the extent of the Pierre Sea during Upper Campanian (Bearpaw) time (Warren and Stelck, 1958).

The St. Mary River Section

South of Lethbridge, in Lsd. 2, Sec. 7, Tp. 7, R. 21, W 4th Mer. (figure 3) the Oldman-Bearpaw contact is well exposed where it forms the south bank of the St. Mary River. The section studied is a fresh-appearing outcrop on the outside, undercut bank of a meander.

For approximately 50 feet above river level the section is composed of shale, coal, and minor thin siltstone stringers. The shale is dark grey to greyish brown, silty to plastic and unctuous, highly carbonaceous for the most part,



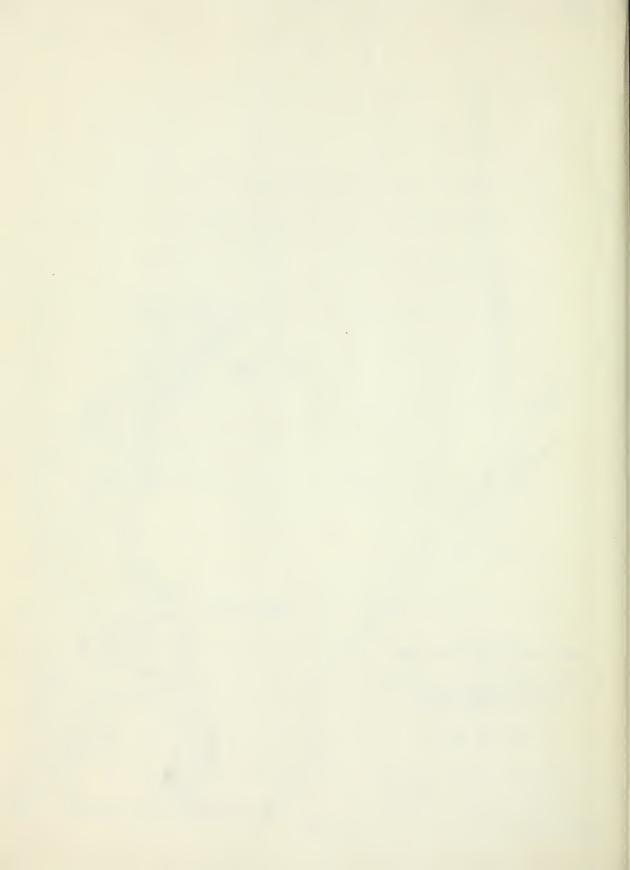
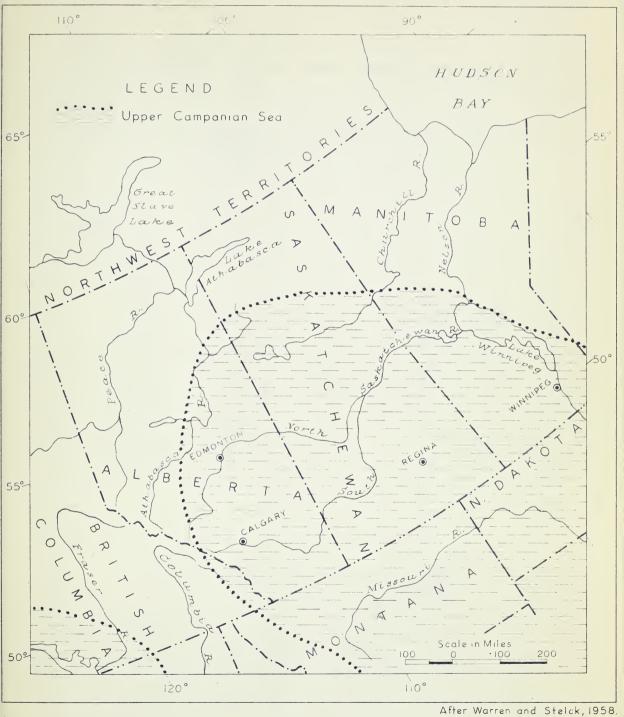
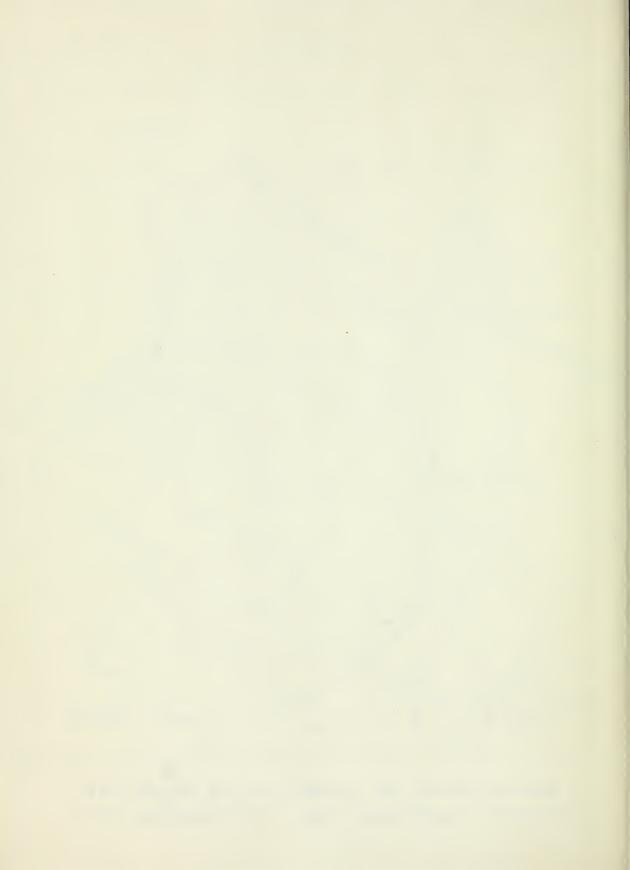


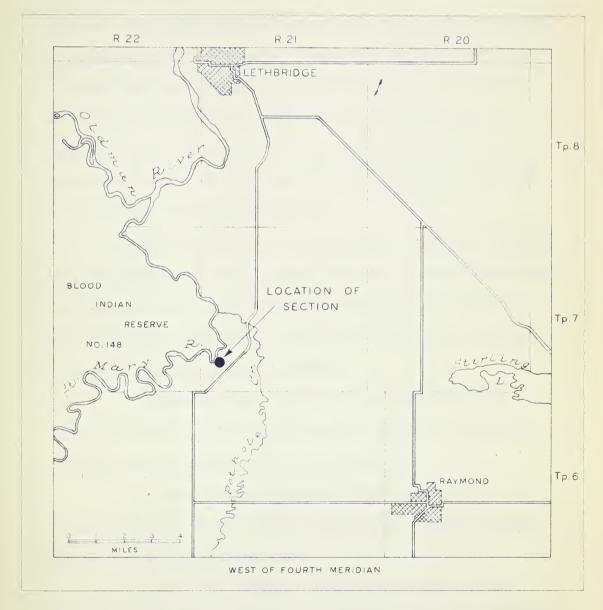
FIGURE 2

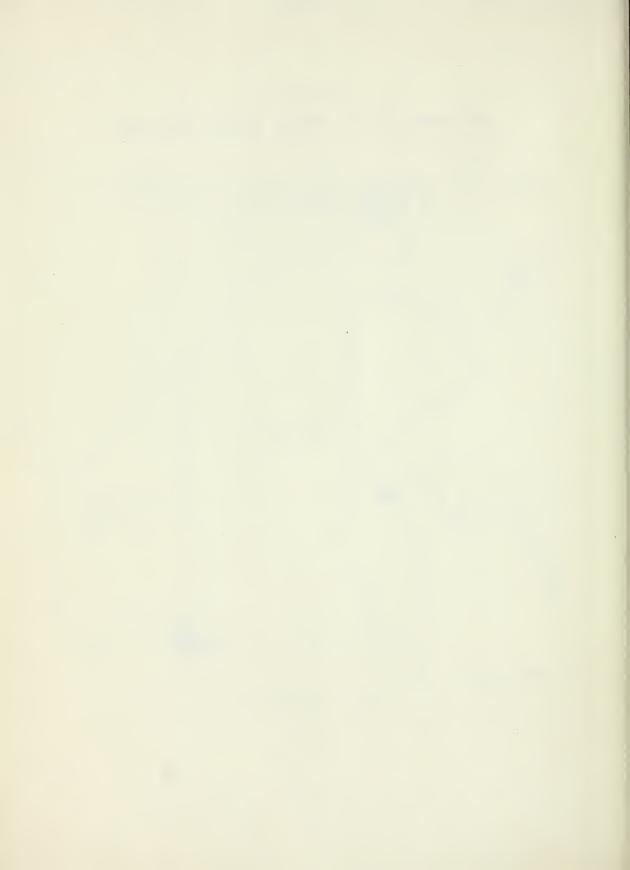


MAP SHOWING THE EXTENT OF THE PIERRE SEA IN UPPER CAMPANIAN TIME (THE BEARPAW SEA).



LOCATION OF ST. MARY RIVER SECTION





with abundant fragmental plant remains. In places a shaly habit has developed but usually these shales are earthy or blocky. Coal occupies about fifteen feet of the total section, the thickest seams being five feet and four feet, the remainder occurring in seams from a fraction of an inch to a foot or two in thickness. Some of the highly carbonaceous shales could be termed sub-coals. A thin (0.01 ft.) seam of bentonite occurs above a two-foot coal seam about 10 feet above the base of the section. Above this upper coal seam there is a 10-foot bed of dark grey to brown, fissile, silty shale, which is very silty in thin beds and has a thin (0.4 ft.) concretionary layer running through it.

The beds overlying this horizon are typical of the Bearpaw, the shales being soft, greyish brown, blocky to earthy, and uniform. Thin hard, discontinuous, concretionary zones are common and the first bentonite bed occurs 106 feet (stratigraphic) above the river level. Gypsum crystals can be seen in abundance on the weathered surfaces of the shale fragments.

The outcrop has a distinct bedded or banded appearance below the upper coal seam but, except for the concretionary layers and bentonite beds, the Bearpaw portion of the section has a massive appearance.

The contact of the two formations has been picked at either the top of the upper coal seam (Dowling, 1917) or at the top of the silty shale overlying the upper coal (Russell and Landes, 1940).

In order to fix the boundary of the non-marine-marine contact as closely as possible, Mr. J. H. Wall, Research Council of Alberta, examined the

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microfauna of the samples collected by the author. On this evidence Mr. Wall placed the contact at the top of the 10-foot silty shale overlying the upper coal (figure 4). This shale is the highest horizon in the section carrying plant remains.

Sample Collecting

The section was measured with a Jacob's staff and samples were collected every two feet. Relatively fresh samples were obtained by digging two feet into the outcrop before collecting the sample. Forty-five samples were collected over the 95 feet of section that was measured and described.

Results of Analysis

The minus two micron size fraction was separated from 44 of the samples and analysed for SiO_2 , Al_2O_3 , Fe_2O_3 , TiO_2 , P_2O_5 , CaO, MgO, K_2O , Na $_2O$ and loss on ignition.

A technique of boiling and stirring was used to get the clays in suspension in order to perform the size-separation by sedimentation. The plus 2 micron size fraction was removed so that the variations in sand and silt content would not result in meaningless SiO₂ values that would obscure minor variations in the less abundant constituents. No chemicals were used in disintegration of the samples or dispersion of the clays because it was thought that the addition of chemicals might cause some incalculable change in the clay-chemistry. Samples of oriented clay aggregates were prepared on glass slides for x-ray analysis. The details of the method of sample preparation are given in Appendix A.

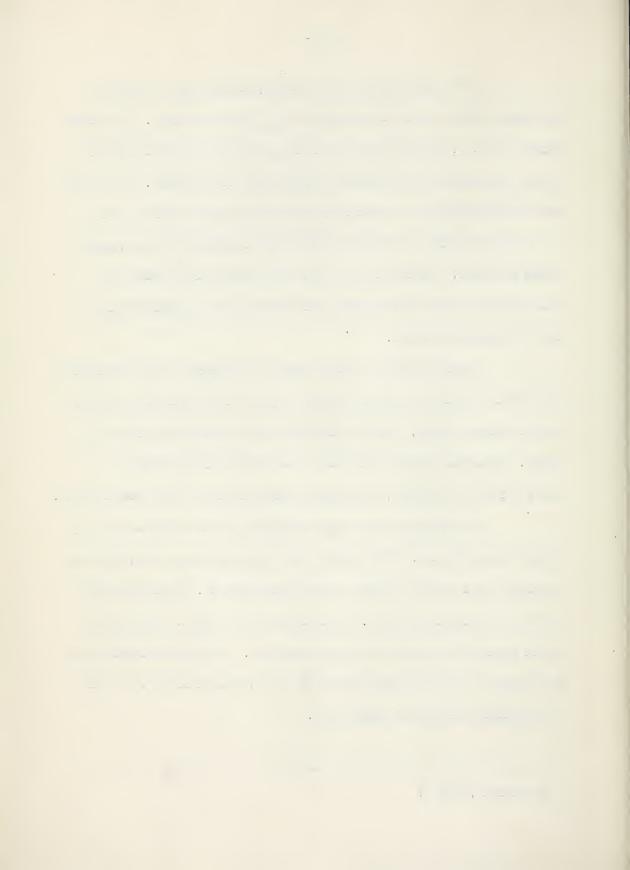
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The methods used in chemical analysis were those of Brannock and Shapiro (1956) except in the analysis of Al_2O_3 and the volatiles. The method outlined by M. L. Jackson was used for Al_2O_3 , after it was found that results obtained using Brannock and Shapiro's method were not reproducible. The samples were ignited at 950°C in an electric furnace to obtain loss-on-ignition since it was thought that little value would be derived by determining total water, organic matter and sulfur. No attempt was made to differentiate between ferric and ferrous iron since the results might be more indicative of a weathering factor than of a depositional factor.

The rapid-silicate-analysis method was adopted for this investigation in an effort to determine whether chemical analysis of rocks, reliable enough for special geologic studies, could be obtained this way by personnel of technical status. The results indicate that except in very special studies involving a series of rocks and analyses, conventional methods likely give more useful results.

No attempt has been made to determine the error involved but every analysis was duplicated. Where the ${\rm SiO}_2$ and ${\rm Al}_2{\rm O}_3$ percentages were reproduced to within one per cent the analysis was considered accurate. In most cases the results were reproduced within 0.5 per cent for ${\rm SiO}_2$ and ${\rm Al}_2{\rm O}_3$. Considerably greater accuracy was obtained for other constituents. The flame photometer used in potassium and sodium determinations give high values for sodium. The error is consistent and results are reproducible.

See footnote, page 6.



Sample 13 was analysed by the University of Minnesota Rock

Analysis Laboratory. The results obtained were compared with the author's

analysis in table one below.

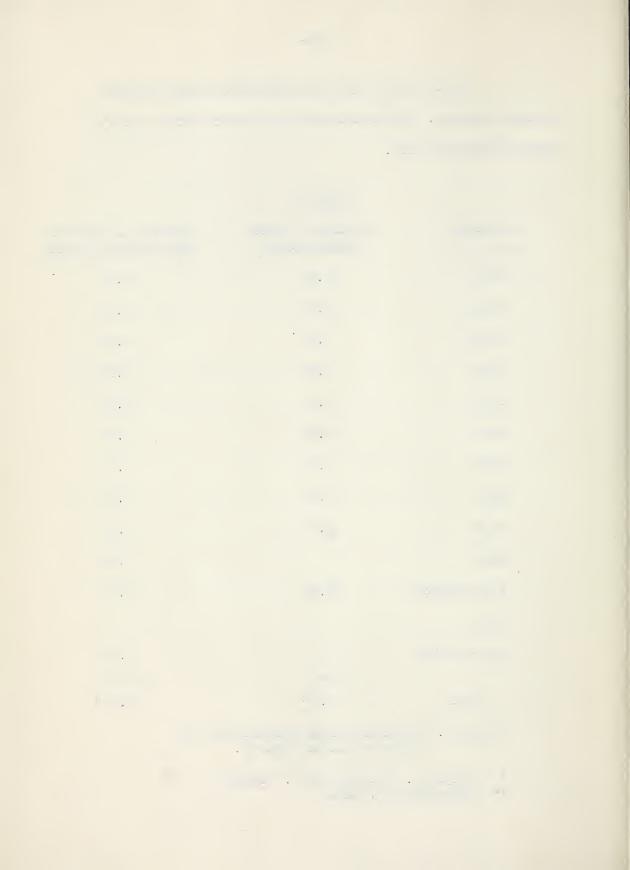
Table One

Constituent	University of Alberta Rapid Analysis	University of Minnesota Rock Analysis Laboratory
SiO ₂	51.99	51.45
Al ₂ O ₃	17.76	17.75
Fe ₂ O ₃	5.03	4.65
${ m TiO}_2$	0.39	0.58
P ₂ O ₅	0.37	0.18
CaO	0.81	1.09
MgO	1.62	1.69
к ₂ 0	2.26	2.19
Na ₂ O	2.71	2.15
MnO		0.04
Loss on ignition	14.83	15.45
H ₂ O +		
H ₂ O - (110°C)		2.76
Total	97.77*	99.98**

Table One. Comparison of rapid analysis results with a check analysis for sample 13.

^{*} Analysts H. Wagenbauer and R. Farvolden

^{**} Analyst Eileen H. Oslund



Results of "Rapid" Analysis

Table Two

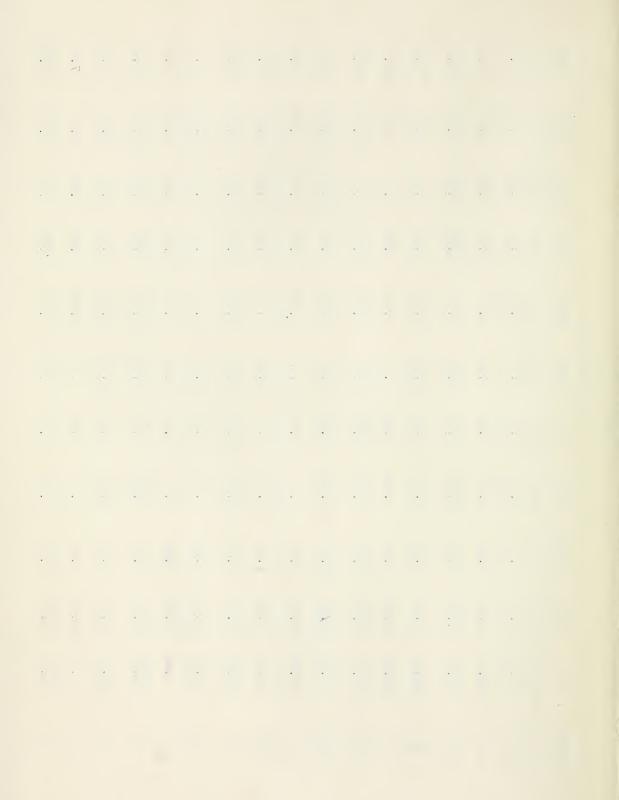
Total	97.65	100.21	97.49	97.54	97.37	18.76	97.35	60.86	98.62	99.44	98.78	97.40	68.86	98.56
lg. Loss	7.50	7.51	7.68	7.39	7.70	7.82	7.83	7.81	7.66	7.77	8.14	7.95	8.20	8.01
Na ₂ O	0.22	0.23	0.19	0.25	1.25	0.20	0.25	0.25	0.75	0.49	0.40	0.44	0.37	0.55
K ₂ O	2.42	2.56	2.66	2.64	2.88	2.96	2.78	2.84	2.90	2.82	3.02	2.83	2.57	2.76
MgO	2.09	2.09	1.65	1.72	1.79	1.68	1.75	1.65	1,55	1.65	1.42	1.25	1.38	1.55
CaO	0.43	0.77	0.77	0.53	0.48	0.58	0.38	0.38	0.38	0.34	0.34	0.58	0.43	0.19
P ₂ O ₅	0.35	0.49	0.33	0.36	0.54	0.43	0.35	0.34	0.42	0.41	0.47	0.44	0.39	0.30
TiO2	0.53	0.54	09.0	0.58	0.57	0.57	0.61	0.62	0.59	0.61	0.50	0.59	0.50	0.57
Fe ₂ O ₃	6.88	8.09	6.63	7.73	7.24	96.9	7.12	7.37	7.24	7.40	92.9	6.78	8.14	7.32
A1203	19.48	18.96	20.64	20.64	19,71	19.71	20.46	20.16	21.57	21.60	21.51	21.24	21,33	21.60
. SiO ₂	57.75	58.97	56.34	55.70	56.21	57.00	55.82	56.67	55.56	56.35	56.22	55.30	55.58	55.71
Sample No. SiO_2	47	46	45	44	43	42	41	40	39	38	37	36	35	34

Sample No. SiO_2	SiO_2	Al ₂ O ₃	Fe ₂ 0 ₃	TiO2	P ₂ O ₅	CaO	MgO	K ₂ O	Na ₂ O	lg. Loss	Total
33	57.08	21.63	5.20	0.45	0.15	0.58	1.22	2.77	0.55	7.88	98.51
32	55,91	22.49	6.42	0.58	0.27	0.24	1.59	2.24	0.35	86.8	20°66
31	55.38	21.12	7.73	0.56	0.28	0.67	1.89	2.29	0.43	8.61	96.86
30	55.38	21.12	6.83	0.61	0.20	0.48	2.16	2.40	0.53	8.53	98.24
29	55.98	20.88	96.9	0.58	0.22	0.57	1.89	2.44	99.0	8.25	98.43
28	56.64	21.12	7.02	0.58	0.19	0.48	1.79	2.37	76.0	7.64	08.80
27	57.29	21.84	5.49	0.67	0.21	0.48	1.65	2.31	1.14	7.49	98.57
26	58.14	19.32	5.73	0.53	0.22	0.57	1.69	2.14	2,27	7 .05	99.76
25	60.82	18.96	5,41	0.52	0.22	0.48	1.45	2.15	2.20	6.45	99°86
24	56.45	20.76	5.91	0.56	0.18	0.38	1.86	2.39	2.18	8.39	90.66
23	57.43	21.24	5.69	09.0	0.20	0.38	1.75	2.42	2.00	7.61	99.32
22	56.97	20.70	5.96	0.57	0.24	0.57	1.86	2.54	1.82	7.67	06.86
21	54.59	19.80	9.12	0.48	0.38	0.48	1.18	2.12	0.51	8.81	97.47
20	56.48	20.88	5.30	0.54	60.0	0.57	1.89	2.40	1.06	8.74	97.95
19	57.71	21.12	5.45	0.56	0.13	29.0	1.55	2.45	1.63	7.23	98.50

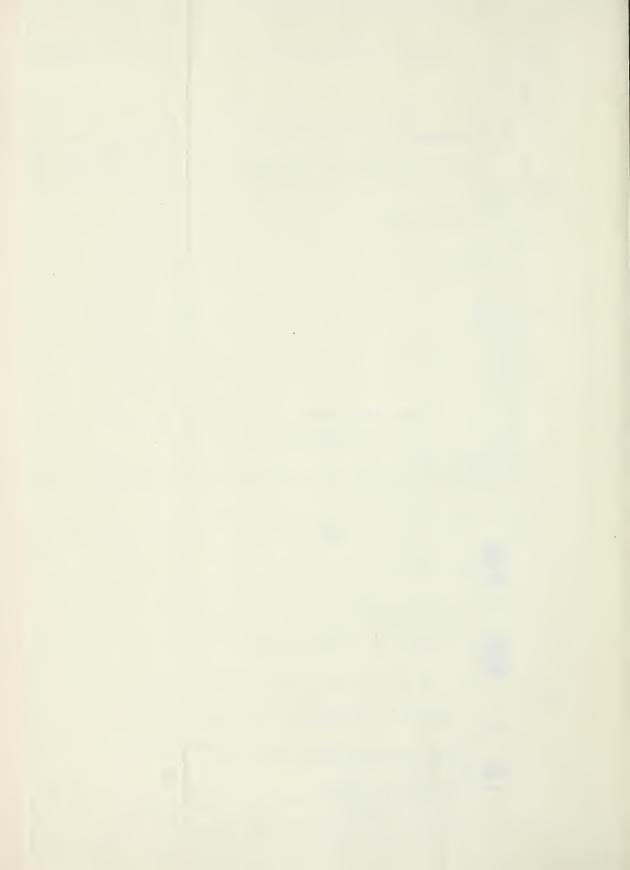
Table Two - 2

Sample No.	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	P ₂ O ₅	CaO	MgO	K ₂ 0	Na ₂ O	lg. Loss	Total
18	56.15	19.74	7.11	0.56	0.24	0.72	1.72	2.19	2.01	7.55	66.76
17	58.17	19.08	4.60	09.0	0.19	0.72	1,55	2.17	2.58	7.98	97.64
16	56.41	21.48	5.36	0.61	0.26	0.53	1.79	2.35	1.37	8 .00	98.16
15	55.24	21.24	5.47	0.59	0.17	0.48	1.55	2.09	2.18	66°8	00°86
14	55.24	19,56	5.19	0.46	0.20	0.91	1.45	1.66	3.12	9.92	97.71
13	51,99	17.76	5.03	0.39	0.37	0.81	1.62	2,26	. 2.71	14.83	77.79
12	48,55	17,76	13.40	0.29	0.41	1.15	2.30	1.81	2.01	10.73	98.41
11	54.59	20.28	5.43	0.55	0.21	0.76	2.23	1,85	1.78	08.6	97.48
01	56.53	19.80	5,11	0.56	90.0	92.0	1.72	2.22	1.68	12.25	100.69
6	56.08	19.20	5.38	0.62	0.18	0.76	2.09	1,73	1.87	8.84	96.75
80	53.23	20.40	5.43	0.42	0.15	0.91	1.79	1.81	1.75	12.30	98.19
9	55.20	19.20	5.89	0.57	0.11	0.67	2.19	2.23	1.60	8.43	60.96
ın	50.02	21.48	5.10	0.48	0.01	98.0	1.86	1.82	1.93	10.01	94.17
4	56.45	19.44	5.79	0.61	0.13	92.0	2.06	2.50	1.53	8.26	87.53
က	54.15	21.72	4°64	0.25	90.0	1.05	2.49	1.09	2.68	91.6	97.29
7	53.95	16.56	4°44	0.02	0.12	1.33	2.06	2.50	1.45	14.22	96.65

Table I WO - 0



FOOTAGE FROM	SAMPLE NO. LITHOLOGY FIELD LOG	FIGURE 4 SAMPLE DESCRIPTION			GRA	PHI	CAL	FI	GURE 5. SENTATI	ON OF A	NALYSES		
		Bentonite bed	Na ₂ O	K ₂ O N	1g0	CaO %	P ₂ O ₅	TiO2	Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	Loss on Ignition	K ₂ O/N _{a2} O
100 -		Shale; brown, blacky to earthy, soft, uniform.	1 2		1 2	1 2	0.5	0.5	6 8 10 12	1416 1820 22	% 50 54 58	6 8 10 12 14	0 5 10 15
90 -	46 000	Concretionary layer											
	43 42 41	Shale; brown, blocky to earthy, soft, uniform.	-			-							
80-	39 38 37 000	Concretionary layer; rusty.				The state of the s							contact in of all.)
70 -	36	Shale; brown, blocky to earthy, soft, uniform.	-										marine minatio
j	33 32 31 30 = =	Shale; with thin interbeds of hard siltstone. Shale; brown, soft, micro-micaceous, plant					- - -						ne-non- don exa
	29 = = = = = = = = = = = = = = = = = = =	fragments, minor gypsum, silty at bottom to fisile at top. Siltstone; brown, earthy to shaly, coarse at bottom				-							Mari
50-	25 000	to fine at top, micaceous, glauconitic(?)				-							
	22	Shale; grey, soft, gypsum, blacky to earthy. Coal; slumped Shale; brown, earthy, carbonaceous.				-		,					
	20	Coal. Shale; grey, silty, hard, fissile to earthy, iron-stained, gypsum, plant fragments.				-							
30-	17	Siltstone and shaly siltstone; brown, hard, laminated, grades up into shale. Shale; grey, soft, fişile, a few plant fragments. Coal.			=				(-
20-	15	Shale, grey, brown, soft, blocky to earthy, gypsum, silty. Shale: greyish brown fissile to blacky silty, micro-				- ·	_ _ 						
	12	Coal.					_			1			
10 -	8	carbonaceous, with coal stringers. Bentonite; cream, soft (O.I'). Coal; blocky. Shale; grey, soft, finely blocky. Coal; with minor fissile shale. Shale; medium grey and brown, fissile to finely blocky, silty beds, coal fragments. Silty ane.							_				
0 -	3 2	Shale; medium grey and brown, tissile to tinely blocky, silty beds, coal fragments. Silty zane. Shale, dark-brown, silty, fissile, compact, coal fragments, fassiliferous, (Pelecypod fragments).					-		-				



The totals obtained for most of the analyses are 1. to 2.5 per cent low. These results are within the limits accepted by investigators using this "rapid-analyis" method. The general tendency of the totals to be low is attributed to a systematic error in the loss-on-ignition results. It is thought that the ignited sample, which should be an excellent dessicant, had adsorbed water before weighing, and thus this value is regularly low.

Discussion of Results

The most prominent feature of the analyses is the remarkable constancy of the chemical composition of clay-size fraction of the shales. There are no significant variations in the series except in the case of $\rm Na_2O$, $\rm K_2O$, $\rm P_2O_5$, and total iron.

From consideration of the method of sample preparation (Appendix A) it seems certain that the sodium and potassium of the clays is firmly adsorbed on the basal planes of clay-mineral structures. It is likely that the sodium is associated with montmorillonite and the potassium is associated with illite. Thus the decrease in sodium content from the Oldman to the Bearpaw portion of the section, and the accompanying increase in potassium, can be interpreted as an indication that a portion of the montmorillonite of the ancient continental environment has altered to illite in the marine environment. The ratios of K_2O to Na_2O show this break very well $\frac{figure}{table}$ 3). The ratios cannot be considered as indicative of the relative abundance of mineral species because the K_2O change may be due in part to a degraded illite regaining K.

From the review of the work of other investigators one would expect a distinct change in the MgO content of the samples over the marine -non-marine contact. Since no change was found, it must be assumed that there is no significant variation in the chlorite content of the Oldman and the Bearpaw shales.

The composition of the shales with respect to alumina and silica shows no significant change over the formational contact. This indicates that there is no variation in the amounts of kaolinite present in the two formations and that free quartz is not masking results. The reports of research on Recent sediments indicate that kaolinite is altered in a marine environment. It can be postulated, that since no chemical changes were found which would indicate the alteration of kaolinite, kaolinite was not abundant in the sediments involved.

There is a slight but notable increase in phosphorous toward the top of the section, the increase taking place nearly fifteen feet above the sodium "break", at approximately the same horizon as the potassium "break". There are two factors which could contribute to an increase in phosphorous in the marine portion of the section. Twenhofel (1939) states that phosphorous is transported from the site of weathering in solution as phosphoric acid and as calcium phosphate, the latter being soluble in water containing carbon dioxide and in swamp water rich in organic matter. The nature of the Oldman formation indicates that under conditions that prevailed during deposition the phosphorous in transport could remain in solution and be carried on into the marine environment. There it would be precipitated due to the change in pH and eH. A part of the increase

in phosphorous above the contact could be the result of the greater concentration of animal life in the Bearpaw sea than in the Oldman swamps.

The concentration of total iron shows a very distinct rise from the lower to the upper portion of the section. Iron can occur in the octahedral position in both montmorillonite and illite and is common as a cement or as discrete particles of the oxide. Thus the iron analyses are of no value in interpreting the clay chemistry of the section as there is no way of knowing the nature of the iron occurrence in the rocks. The original ferric and ferrous concentrations in the shale have been altered some indeterminate amount by weathering and therefore ferric-ferrous ratios would be meaningless. However, the iron content has significance as an indicator of environment of deposition. The amount of iron present increases from the non-marine to the marine beds, thus it is likely that the environment during Oldman time was reducing; this condition would allow the iron to be removed by solution as the ferrous ion. Upon entering the more basic waters of the Bearpaw sea the iron would precipitate as the hydroxide. The carbonaceous nature of the Oldman beds indicates that the beds were formed under reducing conditions and this fits the explanation given.

The results are as predicted for Na_2O , K_2O , P_2O_5 and Fe_2O_3 , but not for MgO, Al_2O_3 and SiO_2 . To find an explanation for this variation it is necessary b refer to work related to this investigation.

The mineralogy of the minus two micron fraction of this section has been determined by x-ray analysis (Byrne, P.J.S. and Farvolden, R. N., in preparation). There is a slight, but distinct break in the clay mineralogy at the

contact between the two formations. Chlorite remains essentially constant throughout the interval examined, but montmorillonite, dominant in all samples, is reduced in the Bearpaw shale in favor of illite. These changes are in agreement with the results and interpretation of the chemistry of the samples. The x-ray analysis also reveals the presence of considerable free quartz and amorphous matter and shows the crystallinity of the clays to be generally poor and the crystal sizes small. These last two factors are not what would be expected from the work of other investigators.

Any explanation of the clay-chemistry and mineralogy of these strata must be consistent with those made by Weaver (1958) from his mass of data, which prove that the gross mineralogy of a clay rock depends upon the character of the source material. However, Weaver does not offer sufficient evidence to refute the theory that clays undergo a certain amount of diagenetic alteration. According to Weaver's theory the changes observed in this section at the Oldman-Bearpaw contact necessitate a change in source area.

It is believed that if there was a change in the sediments introduced into the basin, it was due to an increase in volcanic ash fall-out rather than a change in source area.

There are two feasible explanations of the dominance of montmorillonite in all the samples examined. Firstly, montmorillonite can be the
dominant clay mineral in sediments derived from a source area where, due
to insufficient rainfall, there is little leaching of the solum (Milne and Earley,
1958). Secondly, montmorillonite is known to occur in sediments as an alteration

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product of volcanic ash. The montmorillonite in this section was probably supplied by both of these mechanisms.

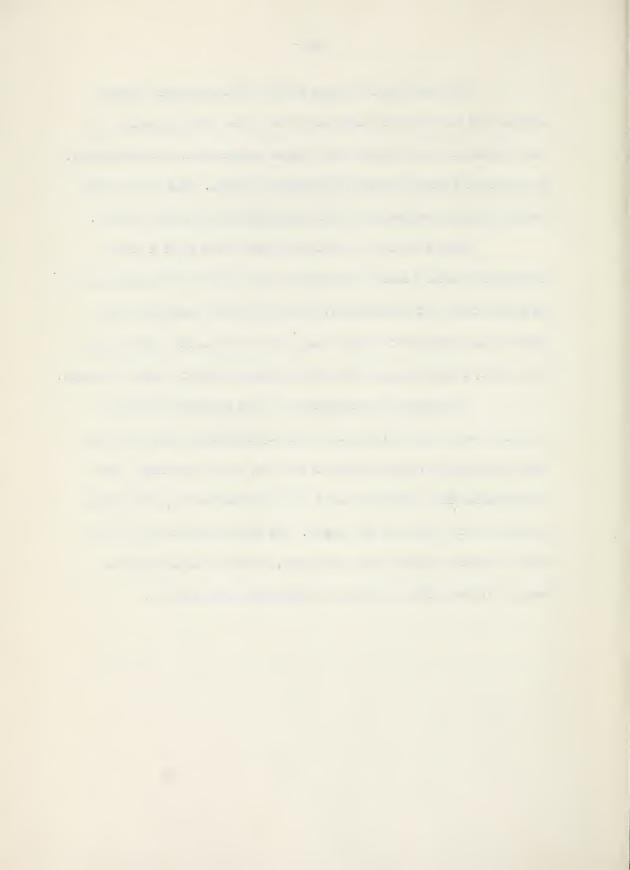
The bentonite beds of the Bearpaw formation and, to a lesser degree, the Oldman formation, are probably altered volcanic ash. The ash would be preserved in distinct beds only where limited circulation prevented its mixing with the sediments brought into the basin by streams. Each of the preserved bentonite beds indicates a fall of ash into the basin, probably over a large area. Previous investigations have shown that the bentonite beds are numerous and are found throughout the Bearpaw section. It is considered that the number of bentonite beds found in any one section does not represent the total ash fall-out. Additional ash fall-outs took place - as proven by bentonite beds at other horizons in other sections - but locally the resulting ash was mixed, by fluctuating currents, with stream-derived muds. Thus a large proportion of the montmorillonite in the Bearpaw shale has a volcanic origin.

An ash fall-out and generation of montmorillonite would tend both to mask and to reduce the effects of diagenesis. Firstly, large quantities of material introduced into the system in this manner would tend to mask the minor changes involved in clay diagenesis. Secondly, the large percentage of montmorillonite would reduce the permeability of the bottom muds which in turn would inhibit diagenesis by removing the muds from contact with the marine environment.

The point might be raised that the present chemistry of these samples does not indicate the chemistry of the shales during deposition and early diagenesis because of chemical changes brought about, after lithification, by interstratal solutions and other late diagenic changes. This does not seem feasible when one considers the impermeable nature of the shales involved.

From the outline on weathering given in this paper it could be argued that the outcrop section examined has been altered and the chemistry is not representative of the buried beds. The shales in the outcrop have been altered to some degree but erosion is very active on the outcrop examined and it is unlikely that the changes have been significant enough to distort the results.

The failure of montmorillonite to alter to chlorite and illite in the marine environment and the lack of good crystallibility in the clays could also be explained by rapid deposition of the beds, so that the bottom muds were prevented from undergoing normal diagenesis because they were quickly removed from the system by deep burial. The shales and coal seams of the Oldman formation and the rather monotonous, massive shale of the lower Bearpaw formation offer no evidence to substantiate this criticism.



Conclusions

- 1. The marine-non-marine contact in the section is reflected in the chemistry of the minus two micron fraction of the shales.
- A considerable portion of the Bearpaw and possibly the Oldman formation
 of this area may be composed of volcanic ash altered to montmorillonite.
- 3. On the basis of the clay-chemistry, the silty shales overlying the upper coal of the Lethbridge member belong to the non-marine sequence.
- 4. The "rapid analysis" method can be used in geologic studies where errors of up to one per cent on the concentrations of the oxides are permissible.

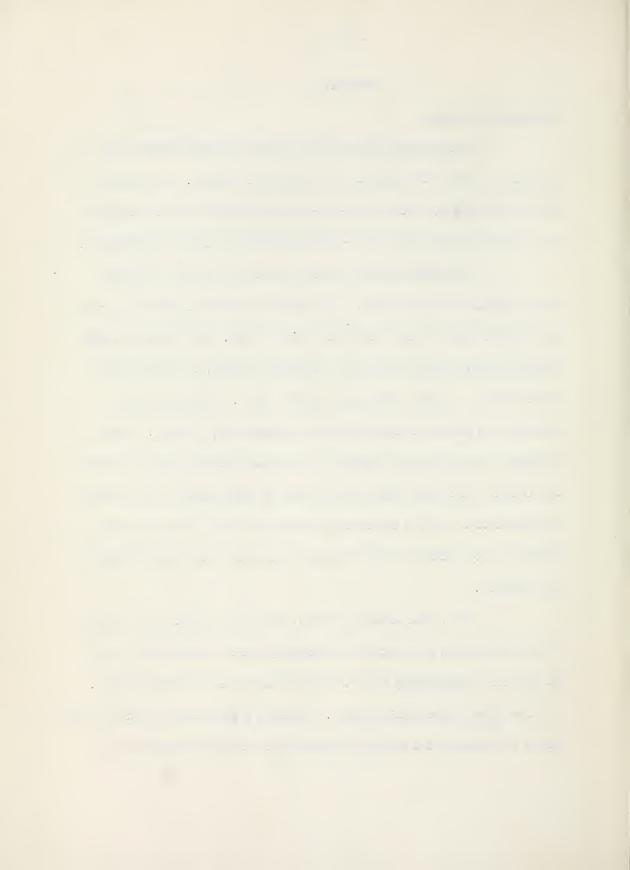
APPENDIX A

Preparation of Samples

Approximately 8 grams of the minus-two-micron fraction was separated from each "raw sample" for the chemical analysis. Two slides of the oriented aggregate of the minus-two-micron fraction of each "raw sample" were prepared on glass slides for x-ray examination for a related investigation.

One hundred grams of the raw sample was crushed in an agate mortar to pass a 60 mesh screen. This crushed material was placed in a 2-litre pyrex beaker which was then filled with distilled water. The mixture was then boiled for approximately 8 hours with intermittent stirring, the volume being maintained by occasional additions of distilled water. The beaker and its contents were allowed to stand and cool for approximately 16 hours. If there appeared to be considerable material in suspension after this period of standing, the contents of the beaker were size-separated by sedimentation. If the sample had flocculated, any clear supernatant liquid was poured off, the volume was brought up with distilled water and the process repeated until a good dispersion was achieved.

When, after standing 16 hours, the beaker contents were observed to be in suspension, the mixture was thoroughly agitated and allowed to stand for 8 hours. Approximately 100 cc. of the suspension from a depth of 10 cm. was then siphoned into another beaker. According to Krumbein and Pettijohn (1938) Stokes law governing the settling of spherical particles can be applied to this



separation, the size fraction removed being minus 2 microns. It was necessary to repeat this separation process three times for almost all dispersions in order to obtain a sufficient quantity of prepared samples for analysis. The large beaker containing the boiled and dispersed mixture was brought up to volume with distilled water for each separation.

The mixture removed by siphoning was evaporated to dryness at 60°C and the residue ground to a fine powder. After drying for ten hours at 100°C the powder was transferred to a dessicator as a prepared sample.

For each sample a portion of the siphoned fluid was drained into a shallow evaporating dish containing two clean glass slides, lying flat, side by side. This fluid was evaporated to dryness, leaving a residue of oriented clay aggragate material forming a film over the glass slides. These samples were used in the x-ray examination of the shales in a related investigation.

Most of the Oldman formation samples formed suspensions with less than four days of boiling. Samples 2 - 15 formed suspensions on stirring. Some of the Bearpaw formation samples required ten days of boiling before they formed suspensions and could be separated.

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Addenda

In these strata there is a good correlation between the concentration of Na₂O and the environment of deposition. However, the marine samples, relatively low in Na₂O, are also the samples which required considerable boiling before going into suspension. It was thought that the sodium in these samples may have been leached by this prolonged boiling. The low Na₂O values might, therefore, be only an index of this prolonged boiling.

Samples 36, 37, 38 were used to check this possibility. One hundred grams of the samples were crushed to pass a 60 mesh screen and then placed in 2-litre pyrex beakers. The beakers were filled to a mark with distilled water and then boiled 8 hours a day for 16 days, the volume being kept up by additions of distilled water. Each night the samples were allowed to cool and flocculate and each morning a sample of the clear supernatant liquid was collected. These daily samples were then analyzed for Na₂O and K₂O (table 3).

Another 2-litre pyrex beaker was filled with distilled water and given the same treatment for 10 days. Daily samples were taken and analyzed for $\rm Na_2O$ (table 3).

It is obvious that the ${\rm Na_2O}$ values in the water increase as boiling progresses. It appears that most of this ${\rm Na_2O}$ is leached from the glassware and the remaining quantities are insignificant.

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